

# Anisotropy in transport and magnetic properties of $\text{K}_{0.64}\text{Fe}_{1.44}\text{Se}_{2.00}$

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We report a study of anisotropy in transport and magnetic properties of  $\text{K}_{0.64}\text{Fe}_{1.44}\text{Se}_{2.00}$  single crystals. The anisotropy in resistivity is up to one order of magnitude between 1.8 K and 300 K. Magnetic susceptibility exhibits weak temperature dependence in the normal state with decrease in temperature with no significant anomalies. The lower critical fields  $H_{c1}$  of  $\text{K}_{0.64}\text{Fe}_{1.44}\text{Se}_{2.00}$  are only about 3 Oe and the anisotropy of  $H_{c1,c}/H_{c1,ab}$  is about 1. The critical currents for  $H\parallel ab$  and  $H\parallel c$  are about  $10\text{-}10^3\text{ A/cm}^2$ , smaller than in iron pnictides and in  $\text{FeTe}_{1-x}\text{Se}_x$  and nearly isotropic.

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## I. INTRODUCTION

Superconductivity discovery in  $\text{LaFeAsO}_{1-x}\text{F}_x$  has triggered intense research activity that resulted in critical temperatures up to 56 K in pnictide materials.<sup>1–5</sup> Soon after, several types of iron-based superconductors have been discovered, such as  $\text{AFe}_2\text{As}_2$  (A = alkaline or alkaline-earth metals, 122-type),<sup>6,7</sup>  $\text{LiFeAs}$  (111-type),<sup>8</sup>  $(\text{Sr}_4\text{M}_2\text{O}_6)(\text{Fe}_2\text{Pn}_2)$  (M = Sc, Ti or V, 42622-type),<sup>9,10</sup> and  $\alpha\text{-PbO}$  type  $\text{FeSe}$  (11-type)<sup>11</sup> etc. The 11-type materials  $\text{FeSe}$ ,  $\text{FeTe}_{1-x}\text{Se}_x$ ,<sup>12</sup> and  $\text{FeTe}_{1-x}\text{S}_x$ <sup>13</sup> provided an example of iron based superconductivity in a rather simple crystal structure without the charge reservoir layer. Yet, these simple binary structures share a square-planar lattice of Fe with tetrahedral coordination and similar Fermi surface topology with other iron-based superconductors.<sup>14</sup> Furthermore, 11-type superconductors contain some distinctive structural and physical features, such as interstitial iron  $\text{Fe}_{1+y}\text{Te}$  and the significant pressure effect.<sup>15–17</sup> Under external pressure, the  $T_c$  can be increased from 8 K to 37 K and the  $dT_c/dP$  can reach 9.1 K/GPa, the highest increase in all iron-base superconductors.<sup>17</sup> This behavior may be understood from the observation related to the anion height between Fe and As (or Se, Te) layers. There is an optimal distance around 1.38 Å with a maximum transition temperature  $T_c \simeq 55\text{ K}$ .<sup>18</sup> The anion height in  $\text{FeSe}$  decreases gradually with the pressure increase towards the optimal value thereby increasing  $T_c$ .<sup>18</sup> Quite importantly, high upper critical fields and currents were demonstrated in iron based superconductors.<sup>19–21</sup>

Another method for tuning of the anion height is the intercalation between  $\text{FeSe}$  layers that can change both the local environment of Fe-Se tetrahedron and the average crystal structure. The intercalation could also decrease dimensionality of conducting bands. The presence of low energy electronic collective modes in layered conductors helps screening Coulomb interaction, which may contribute constructively to superconductivity.<sup>22</sup> This is seen in iron based superconductors: the  $T_c$  increases from 11-type to 1111-type. Very recently the  $T_c$  was raised in iron selenide material to about 30 K by intercalating K, Rb, Cs, and Tl between the  $\text{FeSe}$  layers ( $\text{AFeSe}$ -122

type),<sup>23–29</sup> as opposed to pressure. The intercalation of alkaline metals decreases Se height,<sup>28</sup> and changes the average space group from  $P4/nmm$  of  $\text{FeSe}$  to  $I4/mmm$  of  $\text{AFeSe}$ -122 type. The Fe-Se interlayer distances are expanded and may contribute to electronic and magnetic dimensionality reduction. Furthermore, insulator-superconductor transition (IST) can be induced by tuning the Fe stoichiometry in  $(\text{Ti}_{1-x}\text{K}_x)\text{Fe}_{2-y}\text{Se}_2$  ( $0 \leq x \leq 1$ ,  $0 \leq y \leq 1$ ).<sup>29</sup> This suggests that the superconductivity of  $\text{AFeSe}$ -122 type is in proximity of a Mott insulating state.<sup>29</sup> Thus it is of interest to study electronic and magnetic anisotropy in normal and superconducting states in  $\text{AFeSe}$ -122 in order to shed more light on the superconducting mechanism and possible symmetry of the order parameter.

In this work, we report the anisotropy in electronic transport and magnetization in the normal state of  $\text{K}_{0.64(4)}\text{Fe}_{1.44(4)}\text{Se}_{2.00(0)}$  single crystals. We also present anisotropic parameters of the superconducting state.

## II. EXPERIMENT

Single crystals of  $\text{K}_x\text{Fe}_2\text{Se}_2$  were grown by self-flux method with nominal composition  $\text{K}_{0.8}\text{Fe}_2\text{Se}_2$ . Prereacted  $\text{FeSe}$  and K pieces (purity 99.999%, Alfa Aesar) were put into the alumina crucible, and sealed into the quartz tube with partial pressure of argon. The quartz tube was heated to 1030 °C, kept at this temperature for 3 hours, and then slowly cooled to 730 °C with 6 °C/hour. Plate-like crystals up to  $5 \times 5 \times 1\text{ mm}^3$  can be grown. X-ray diffraction (XRD) spectra were taken with Cu  $K_\alpha$  radiation ( $\lambda = 1.5418\text{ Å}$ ) using a Rigaku Miniflex X-ray machine. The lattice parameters were obtained by fitting the XRD spectra using the Rietica software.<sup>30</sup> The elemental analysis was performed using an energy-dispersive x-ray spectroscopy (EDX) in an JEOL JSM-6500 scanning electron microscope. Electrical resistivity  $\rho(T)$  measurements were performed in Quantum Design PPMS-9. The in-plane resistivity  $\rho_{ab}(T)$  was measured using a four-probe configuration on rectangularly shaped and polished single crystals with current flowing in the ab-plane of tetragonal structure. The c-axis resistivity

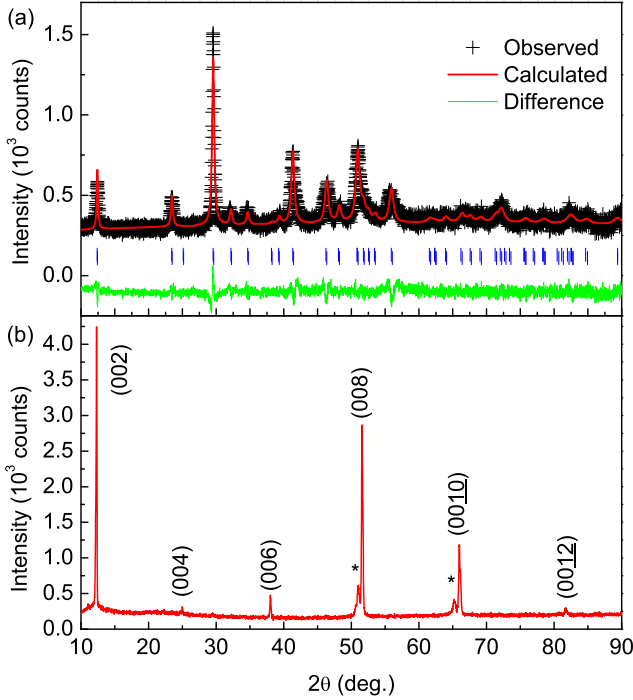


FIG. 1. (a) Powder and (b) single crystal XRD patterns of  $K_xFe_{2-y}Se_2$ , respectively.

$\rho_c(T)$  was measured by attaching current and voltage wires on the opposite sides of the plate-like sample.<sup>31,32</sup> Since the sample surface is easily oxidized, sample manipulation in air was limited to 10 minutes. Sample dimensions were measured with an optical microscope Nikon SMZ-800 with 10  $\mu m$  resolution. Electrical transport and heat capacity measurements were carried out in PPMS-9 from 1.8 to 300 K. Magnetization measurements were performed in a Quantum Design Magnetic Property Measurement System (MPMS) up to 5 T.

### III. RESULTS AND DISCUSSION

Fig. 1(a) shows the X-ray diffraction (XRD) results of the ground crystal. It confirms phase purity with no extrinsic peaks. The powder pattern can be indexed in the I4/mmm space group with fitted lattice parameters  $a = 0.39109(2)$  nm,  $c = 1.4075(3)$  nm ( $R_p = 2.272$  and  $R_{wp} = 3.101$ ), consistent with reported results.<sup>23–25</sup> On the other hand, XRD spectra of a single crystal reveal that the crystal surface is normal to the  $c$  axis with the plate-shaped surface parallel to the  $ab$ -plane (Fig. 1(b)). Furthermore, there is another weak series of (001) diffraction peaks (labeled by the asterisks) associated with the main peaks. These two distinct sets of reflections could arise from the inhomogeneous distribution of K atoms, which also observed in other AFeSe-122 single crystals.<sup>34,35</sup> The average stoichiometry was determined from EDX by examination of multiple points on the crystals. The measured compositions are  $K_{0.64(4)}Fe_{1.44(4)}Se_{2.00(0)}$  (noted as

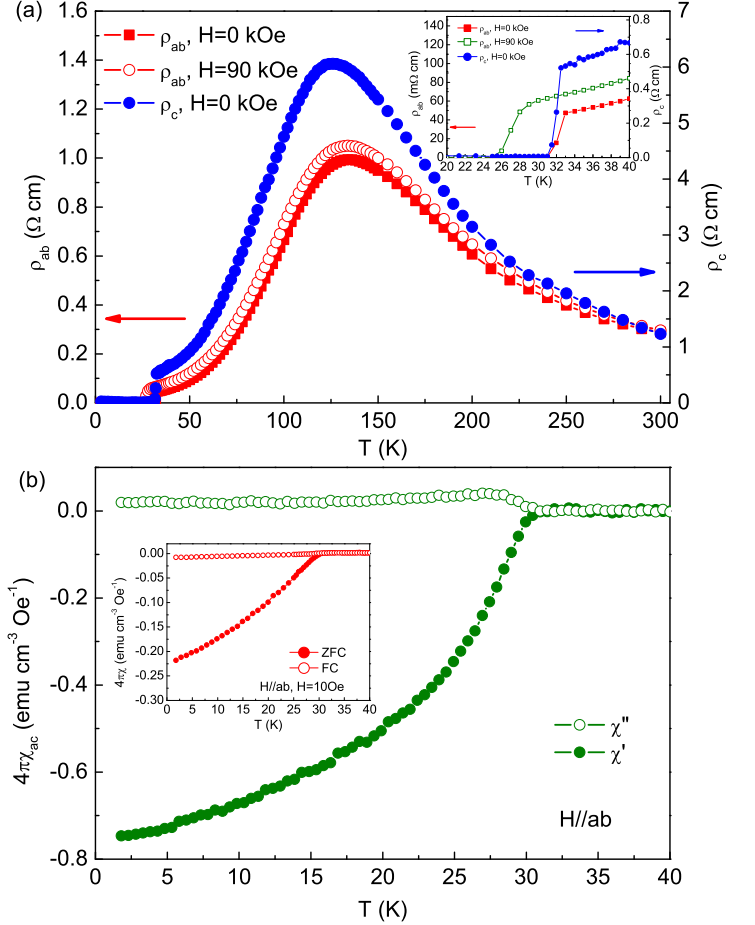


FIG. 2. (a) Temperature dependence of the resistivity  $\rho_{ab}(T)$  and  $\rho_c(T)$  of  $K_xFe_{2-y}Se_2$  with and without  $H=90$  kOe along  $c$  axis. Inset: enlarged resistivity curve near  $T_c$ . (b) Temperature dependence of ac magnetic susceptibility of  $K_xFe_{2-y}Se_2$  in  $H_{ac}=1$  Oe. Inset: temperature dependence of dc magnetic susceptibility with ZFC and FC.

$K_xFe_{2-y}Se_2$ ), indicating substantial with of formation and the existence of K and Fe vacancies. We also measured the composition mapping using EDX. The results exhibit that the spatial distribution of K, Fe, and Se are homogenous.

The main panel of Fig. 2(a) shows the temperature dependence of resistivity in zero field from 1.9 K to 300 K for current along  $ab$  plane and  $c$  axis. At higher temperatures, both  $\rho_{ab}(T)$  and  $\rho_c(T)$  show a metal-insulator transition with a maximum resistivity at about 135 K and 125 K, respectively. The resistivity maximum ( $\rho_{max}$ ) might be related to a scattering crossover arising from a structure or magnetic phase transition and is a typical behavior in AFeSe-122 system.<sup>23–29</sup> Furthermore, the temperature dependence of the  $\rho_{max}$  depends on the extent of iron deficiency in the crystals.<sup>33</sup> From our resistivity data in the normal state below 300 K, the ratio  $\rho_c/\rho_{ab}$  is about 4-12. The anisotropy is much smaller than in  $(Tl,K)_xFe_{2-y}Se_2$  and  $(Tl,Rb)_xFe_{2-y}Se_2$  system, where  $\rho_c/\rho_{ab} = 70-80$  and  $30-45$ .<sup>29,34</sup> On the other hand,

both of  $\rho_{ab}(T)$  and  $\rho_c(T)$  undergo a very sharp superconducting transition at  $T_{c,onset} = 33$  K, shown in the inset of Fig. 2(a). At 90 kOe, the resistivity transition width is broader and the onset of superconductivity shifts to 28 K. However, the  $\rho_{max}$  curve has no obvious shift in magnetic field up to 90 kOe for current transport along both crystallographic axes.

Fig. 2(b) shows the temperature dependence of the ac susceptibility of  $K_xFe_{2-y}Se_2$  single crystal with  $H||ab$ . A clear superconducting transition appears at  $T = 31$  K. This is consistent with the resistivity results. The superconducting volume fraction is about 75% at 1.8 K, indicating the bulk superconductivity in the sample. The broad transitions in  $\chi'$  and  $\chi''$  point to microscopic inhomogeneity. Inset in Fig. 2(b) shows the dc magnetic susceptibility for  $H||ab$  with zero-field cooling (ZFC) and field cooling (FC). Diamagnetism can be clearly observed in both measurement and the  $T_{c,onset}$  is almost the same as that determined from the ac susceptibility. On the other hand, the magnetization measured with FC is very small, which is a common behavior in two-dimensional superconductors, such as  $(Pyridine)_{1/2}TaS_2$ ,<sup>36</sup> and  $Ni_xTaS_2$ .<sup>37</sup> The small magnetization values for FC is likely due to the complicated magnetic flux pinning effects in the layered compounds.<sup>36</sup>

Temperature dependence of magnetic susceptibility in the normal state is shown in Fig. 3(a) for  $H||ab$  and  $H||c$  with  $H = 1$  kOe. A sudden drop at about 30 K corresponds to the superconducting transition. For  $H||c$ ,  $\chi_c$  weakly decreases with temperature below 300 K and exhibits a weak upturn below 120 K. When the magnetic field is in the  $ab$  plane,  $\chi_{ab}$  exhibits similar behavior but the minimum of susceptibility is located at about 175 K. The magnetic susceptibility enhancement with increase in temperature above 200 K is neither Pauli nor Curie-Weiss. It suggests the presence of magnetic interactions. This has not only been observed in other AFeSe-122 compounds,<sup>25,26,29,38</sup> but also in  $BaFe_2As_2$  due to two dimensional short range AFM spin fluctuations.<sup>39,40</sup> The AFM interaction is possibly related to the Fe deficiency and is an intrinsic properties of  $AFe_2Ch_2$  ( $Ch = S, Se$ ).

The initial dc magnetization versus field  $m(H)$  at  $T = 1.8$  K for both directions is shown in Fig. 3(b). The shape of the  $m(H)$  curves points that  $K_xFe_{2-y}Se_2$  is a typical type-II superconductor. The peak in  $m(H)$  is about 2 kOe for  $H||c$ , consistent with the previous report.<sup>23</sup> However, it should be noted that  $H_{c1}$  is often much smaller than the peak value in  $m(H)$  curve. In  $K_xFe_{2-y}Se_2$ , the  $m(H)$  curve deviates from linearity at much lower field. The enlarged parts are shown in Fig. 3(c) and (d). The  $H_{c1}$  is usually determined by the field where the  $m(H)$  deviates from linear relation.<sup>41</sup> However, small  $H_{c1}$  introduces the significant error, so it is hard to evaluate the  $H_{c1}(0)$  using  $H_{c1}(T) = H_{c1}(0)[1 - (T/T_c)^2]$ . The approximate  $H_{c1,ab}(T = 1.8$  K) and  $H_{c1,c}(T = 1.8$  K) are 3.0(5) Oe.

Fig. 4(a) and (b) show the magnetization loops for  $H||c$  and  $H||ab$  with field up to 50 kOe. The paramagnetic

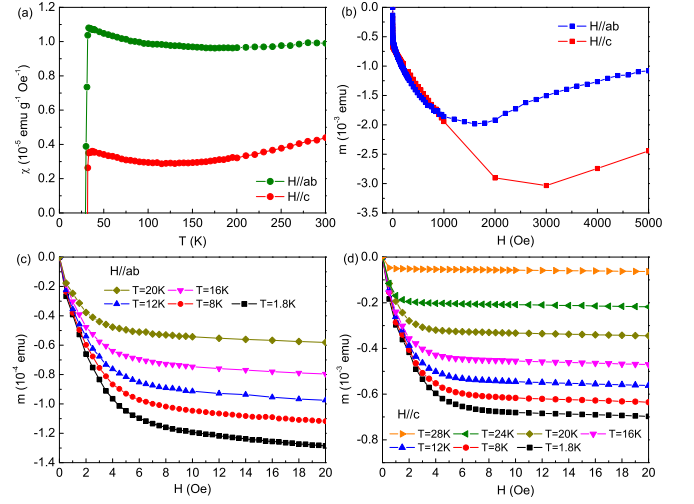


FIG. 3. (a) Temperature dependence of magnetic susceptibility measured at 1 kOe for  $K_xFe_{2-y}Se_2$  crystal with  $H||ab$  and  $H||c$ . (b) Magnetization curve of  $K_xFe_{2-y}Se_2$  at  $T = 1.8$  K for  $H||ab$  and  $H||c$ . (c) and (d) Low field parts of  $m(H)$  at various temperature for  $H||ab$  and  $H||c$ , respectively.

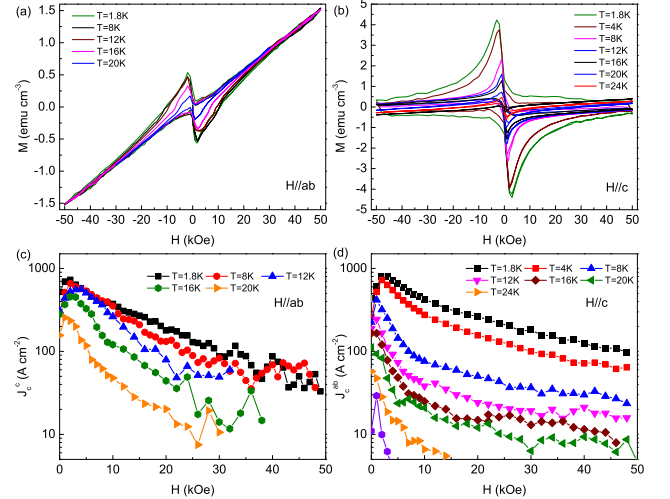


FIG. 4. Magnetization hysteresis loops of  $K_xFe_{2-y}Se_2$  for (a)  $H||ab$  and (b)  $H||c$ . (c) In-plane and (d) interplane superconducting critical currents as determined from magnetization measurements using the Bean model.

background exists for both directions, and is more obvious for  $H||ab$ . This paramagnetic background originates from the non-superconducting fraction. The shapes of  $M(H)$  and  $M(T)$  (Fig. 3) are typical of type-II superconductors with some electromagnetic granularity.<sup>42,43</sup> The critical current is determined from the Bean model.<sup>44,45</sup> For a rectangular-shaped crystal with dimension  $c < a < b$ , when  $H||c$ , the in-plane critical current density  $J_c^{ab}(H)$  is given by

$$J_c^{ab}(H) = \frac{20\Delta M(H)}{a(1 - a/3b)}$$

where  $a$  and  $b$  ( $a < b$ ) are the in-plane sample size in

cm,  $\Delta M(H)$  is the difference between the magnetization values for increasing and decreasing field at a particular applied field value, measured in emu/cm<sup>3</sup>, and  $J_c^{ab}(H)$  is the critical current in A/cm<sup>2</sup>. It should be noted that the paramagnetic background has no effect on the calculation of  $\Delta M(H)$ . The situation is more complex when  $H \parallel ab$ . There are two different current densities: one is the vortex motion across the planes,  $J_c^c(H)$ , and another is parallel to the planes,  $J_c^{\parallel}(H)$ . Usually  $J_c^{\parallel}(H) \neq J_c^{ab}(H)$ . If assuming  $a, b \gg c/3 \cdot J_c^{\parallel}(H)/J_c^c(H)$ ,<sup>45</sup> we can obtain  $J_c^c(H) \approx 20\Delta M(H)/c$ . Magnetic field dependence of  $J_c^c(H)$  and  $J_c^{ab}(H)$  is shown in Fig. 4(c) and (d). It can be seen that the critical current decreases with applied field and the ratio of  $J_c^c(H)/J_c^{ab}(H)$  is approximately 1. The critical current densities for both directions are  $10\text{-}10^3$  A/cm<sup>2</sup>, which is much smaller than those of BaFe<sub>2-x</sub>Co<sub>x</sub>As<sub>2</sub> in the same temperature range.<sup>46</sup>

#### IV. CONCLUSION

In summary, we have presented anisotropic transport and magnetic properties of K<sub>0.64(4)</sub>Fe<sub>1.44(4)</sub>Se<sub>2.00(0)</sub> sin-

gle crystals with  $T_{c,onset} = 33$  K and free of iron impurities. The resistivity anisotropy is much smaller than in other AFeSe-122 compounds. Magnetization decreases in the normal state with decreasing temperature from 300 K, which suggest that the presence of AFM interactions. The lower critical fields  $H_{c1}$  are only about 3 Oe at 1.8 K and the anisotropy of  $H_{c1,c}/H_{c1,ab}$  is about 1. The critical current values are isotropic and is about  $10\text{-}10^3$  A/cm<sup>2</sup> for both directions below 50 kOe.

#### V. ACKNOWLEDGEMENTS

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